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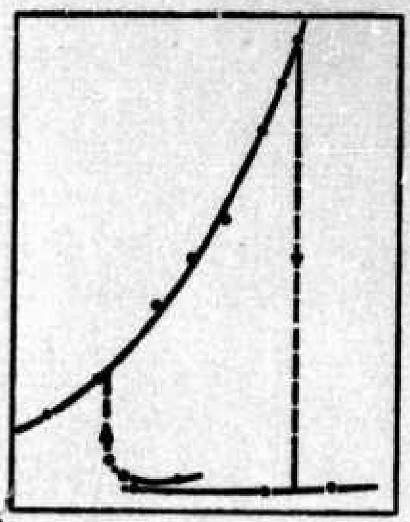

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PROJECT THEMIS

Vibration and Stability of Military Vehicles

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Report No. 21

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"Vibration and Stability of Military and
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ABSTRACT

The objective of this research was to further elucidate the fatigue behavior of commercially pure arc-cast polycrystalline molybdenum; in particular, to investigate the possible relationship between the French damage line and cyclic dependent yield behavior, and to determine coaxing phenomena as a function of grain size. Although the French damage line and the cyclic yield point removal line appear macroscopically similar, the mechanisms proposed for these two phenomena are quite different. The removal of the upper yield point under cyclic stress conditions is explained in terms of localized cumulative multiplication of mobile dislocations, whereas the French damage line is explained in terms of the initiation and propagation of microcracks. Molybdenum, vacuum annealed at 2150 F, 2600 F and 3000 F with resulting ASTM grain sizes of 7-8, 4-5 and 2-3 respectively, was subjected to various coaxing patterns. Using a cycle ratio summation as a criteria, no definite coaxing response pattern occurred for all three molybdenum grain sizes. Cycle ratio summations Σ n/N ranged from .004 to 808. Thus, Molybdenum did respond to coaxing under certain applied stress and cycle increment conditions.

KEY WORDS

Fatigue, Strain Aging, Coaxing, Damage, Overstress, Understress, Grain Size, Cumulative Damage, Crack Initiation, Yielding, Molybdenum.

INTRODUCTION

The interaction of dislocations with interstitial impurity atoms in bcc metals is the primary mechanism which explains the upper yield point, the effect of cyclic stressing on subsequent yield behavior, the sharp knee in the S-LogN diagram and coaxing behavior. This interaction, which controls the above four phenomena, is often integrated into a single concept, simply defined as strain ageing behavior. Cottrell (1), Johnston and Gilman(2) and Hahn (3) have shown the upper yield point is dependent upon this interaction. The upper yield point in iron, low carbon steel and molybdenum has been removed under certain precyclic conditions (4-11). This removal is dependent upon the alternating stress magnitude and number of applied cycles which control the dislocation-interstitial atom interactions. Strain aging models have been used to explain the sharp knee in the S-LogN diagram(12-14). The phenomena of coaxing, which is the resulting increase in fatigue strength or fatigue life caused by progressive understressing, occurred only in materials controlled by strain aging mechanisms (15).

Most of the above mentioned phenomena have been extremely well documented with iron and low carbon steel, whereas other bcc metals containing interstitial atoms of carbon, nitrogen, hydrogen and oxygen have received less attention. Molybdenum, for example, has been shown to possess many of the strain aging dependent characteristics (10,11,16-22), however the total spectrum is still incomplete The objective of this research was to further elucidate three aspects of fatigue behavior of commercially pure arc-cast polycrystalline molybdenum which are macroscopic manifestations of the interaction between dislocations and interstitial impurity atoms: in particular, to investigate the possible relationship between cyclic dependent yield behavior and the French damage line and to determine coaxing phenomena as a function of grain size.

EXPERIMENTAL PROCEDURES AND RESULTS

Cyclic Yield Behavior

Dubensky and Stephens (10) showed the upper yield point in vacuum annealed arc-cast polycrystalline molybdenum could be removed under certain precycling stress conditions. A summary of their work is shown superimposed on an S-LogN diagram in Fig. 1. The solid line represents the mean S-LogN curve obtained under fully reversed axial loading at room temperature. The solid circles represent combinations of fully reversed stresses and applied precycles for different specimens which did not remove the upper yield point in a subsequent tensile test. The squares indicate combinations of fully reversed stresses and applied precycles which did cause removal of the upper yield point in a subsequent tensile test. Note, data points shown do not represent fatigue failures. The dashed line represents the locus of points seperating the different yield behavior. Thus, the upper yield point in molybdenum can be removed under certain combinations of alternating stress and applied cycles. In all cases the stress magnitudes were below the static upper yield point stress but above the fatigue limit.

The locus of points separating the two regions on the S-LogN diagram in Fig. 1 resembles the French damage line obtained in certain materials (23-26). Since additional molybdenum from the same heat was available, an additional investigation was made to compare the yield point removal line with the French damage line.

French Damage Line

The commercially pure arc-cast molybdenum as-received was in the form of 0.00952 m (3/8 in.) diameter cold-drawn rod which had been stress relieved for one hour at 1550 F. The as-received chemical composition is shown in Table 1. Hour glass fatigue specimens with a reduced diameter of 0.0033 m (0.130 in.) were machined and polished with decreasing grades of 0 to 000 emery cloth. The final polishing scratches were in the longitudinal direction to minimize stress

concentraions. After final polishing, specimens were vacuum annealed for 1 hour at 2150 F in a vacuum of 8×10^{-5} to 1×10^{-5} torr. The resultant equiaxed grain size was approximately ASTM 7. All specimens were stored in a desciccater after heat treatment. Tensile yield properties, hardness and grain size were essentially the same as values obtained in the previous investigation and these are listed in column 1 of Table 2.

An S-LogN diagram was obtained at room temperature under fully reversed axial conditions at 30 Hz with the previously used model SF-1-U Sonntag universal fatigue machine. The mean S-LogN curve is shown in Fig. 2 as the solid line. Fatigue data points have not been included since they could perhaps be confused with the damage data points shown. The fatigue limit based upon no failures at 2×10^7 cycles was approximately 258 MN/m^2 (37.5 ksi.).

In determining the French damage line, twenty specimens were subjected to fully reversed axial stresses above the fatigue limit and then retested at slightly below the fatigue limit at 251 MN/m^2 (35 ksi.). If a specimen did not fail within 5×10^6 cycles at the retest stress level, it was assumed that fatigue damage from the initial over stressing did not occur. However, if a fatigue failure occurred at the retest level, it was assumed that initial overstressing did cause fatigue damage. This assumed damage criteria was defined by H. J. French (23).

The magnitude of the overstress and the number of applied cycles for each specimen are superimposed on the S-LogN diagram in Fig. 2. Specimens represented by circles did not fracture within 5×10^6 cycles when retested at 251 MN/m^2 (35 ksi.) after overstressing. It was assumed that beyond 5×10^6 cycles the specimens could withstand a much greater number of cycles without failure. Specimens represented by squares fractured within 5×10^6 cycles when retested at 251 MN/m^2 (35 ksi.). For example, the specimen represented by point A in Fig. 2 was subjected to an overstress of 414 MN/m^2 (60 ksi.) for 2×10^4 cycles and then retested at 251 MN/m^2 (35 ksi.). The specimen fractured in less than 5×10^6 cycles and therefore was considered to have been damaged

by the overstressing. The French damage line is shown in Figure 2 as the broken line. The line would be shifted slightly, if a different retest stress level had been chosen. For example, a lower retest value would tend to shift the line to the right and a higher value would cause a shift to the left. Thus, due to scatter and retest level, the French damage line is slightly variable.

In order to compare the French damage line with the cyclic yield behavior, the two results are superimposed in Fig. 3. Both mean S-LogN curves for the two groups of molybdenum specimens heat treated seperately to identical conditions are shown. It is apparent that the fatigue life of the molybdenum specimens used in the damage experiments is less than that in the cyclic yield behavior study. The fatigue limit, however, for both groups is essentially the same. The difference in fatigue life is primarily attributed to a protective vaseline coating which remained on the specimens during the cyclic yield behavior study. This coating was inadvertantly excluded from specimens in the damage study. As reported by Roberson (21), the fatigue life of molybdenum is affected by oxygen and atmospheric moisture. Normal variation in separate heat treatments is considered a secondary factor It is also apparent that differences exist between the damage line and the cyclic yield behavior line. In particular, the yield point could not be removed with alternating stresses below 379 MN/m^2 (55 ksi.), whereas the damage line existed at stresses down to 226MN/m^2 (40 ksi.).

Coaxing

Orava (17) showed that both the upper and lower yield point stress of polycrystalline molybdenum was dependent upon strain rate and grain size. The yield drop and the yield elongation were substantially reduced in grain size diameter 0.098 mm (ASTM No. 3.5) as compared to a grain size of 0.027 mm diameter (ASTM No. 7.5). Orava's results were in agreement with the Petch equation for relating grain size to the lower yield point stress. This grain size dependence of yielding in molybdenum motivated the investigation for determining coaxing

in molybdenum as a function of grain size.

Tensile and rotating beam fatigue specimens were machined from the previously described heat of arc-cast molybdenum using the same standard machining and polishing procedures as mentioned earlier. Three different groups of specimens were vacuum annealed for one hour at 2150 F, 2600 F and 3000 F. The resulting ASTM grain sizes shown in Fig. 4 were 7-8, 4-5 and 2-3 respectively. The interstitial chemical composition after annealing is shown in Table 3. The carbon content was determined using the Leco Conductometric procedure and the other elements were determined using vacuum fusion techniques. It is seen that the 2600 F heat treatment, obtained in a different furnace than the other heats, absorbed additional carbon and hydrogen. The Rockwell B hardness obtained for each heat is listed in Table 2. The hardness dropped only slightly as the annealing temperature increased. Tensile yield properties were obtained on a Reihle test machine with a constant cross-head speed of approximately 0.127 x 10^6 m/sec. (.03 in./min.). Typical autographic load-deformation curves showing the yield behavior are given in Fig. 5 and average yield values are given in Table 2. The average values are based upon six specimens for 2150 F and two specimens each for 2600 F and 3000 F annealing temperature. Both the upper and lower yield point stresses and the yield drop were reduced for the two larger grain sizes.

All coaxing experiments were performed on a Krouse rotating beam fatigue machine operating between 120 and 150 Hz. Fully reversed bending stresses were computed from the flexure formula. Both constant amplitude fatigue tests and typical coaxing results are shown in Figures 6, 7 and 8. All coaxing data are given in Table 4. The solid circles in Figures 6, 7 and 8 represent constant amplitude fatigue tests which resulted in the mean S-LogN curves shown. The fatigue limits for the three increasing grain sizes were 327 MN/m^2 (47.5 ksi.), 196 MN/m^2 (28.5 ksi.) and 276 MN/m^2 (40 ksi.) respectively.

It is seen in Table 4 that initial coaxing stresses varied from 50 to 95 percent of the fatigue limit with all stress increments at 13.8 MN/m^2 (2 ksi.)

These percentages are also shown in figures 6, 7 and 8. Three different cycle increments of 10^6, 2×10^6 and 10^7 cycles were studied. Column 3 in Table 4 indicates the number of applied cycles at the initial stress level and column 4 indicates the cycle increment at the increased stress levels. The final stress level at failure is given in column 5 and is indicated with an "x" in figures 6, 7 and 8. The cycle ratio summation, Σ n/N, was the primary indication of the effectiveness of the coaxing procedure. An asterisk has been placed adjacent to the cycle ratio summation in Table 4 for those specimens which showed an appreciable effect from the coaxing procedure. The cycle ratio summation ranged from .004 to 808 thus indicating that coaxing response was dependent upon the initial stress level and cycle increment.

Molybdenum annealed at 2150 F and 2600 F had negligible response to coaxing at all initial stress levels when cycle increments of 10^6 or 2×10^6 cycles were applied. Figures 6 and 7 could be misinterpreted in this respect, since stress levels above the fatigue limit were reached before failure. The 10^6 or 2×10^6 cycle increments, however,did not exceed the normal expected fatigue life for a given stress level, and the cycle ratio summation values were less than 6.5 for all cases. 10^7 cycle increments, however, caused appreciable coaxing in the 2150 F group for the 85 percent initial stress level whereas the 95 percent initial stress level did not cause coaxing. The 2600 F group was not subjected to 10^7 increments. The 3000 F group exhibited mixed coaxing response with both high and low initial stresses for both 10^6 and 10^7 cycle increments. For example, at 75 percent initial stress, appreciable coaxing occurred for both 10^6 and 10^7 cycle increments whereas at 87.5 percent initial stress, 10^6 cycle increment causes appreciable coaxing while 10^7 cycle increments caused no coaxing. At 95 percent initial stress, 10^7 cycle increments caused appreciable coaxing whereas 10^6 cycle increments had no coaxing effect.

Fracture surfaces of specimens subjected to coaxing stresses for all three grain sizes are shown in Fig. 9. Some characteristic Fatigue markings can be

identified, but in general all fractures were very crystalline in nature with few fatigue markings, No macroscopic differences could be singled out between constant amplitude fatigue fracture surfaces and coaxed fracture surfaces.

DISCUSSION OF RESULTS

Cyclic Yield Behavior - French Damage Line

A sequence of events occurs during repeated stressing which is identical in the early portion of all cyclic experiments reported in this paper. In the early life of the fatigue process, localized slip occurs as a result of inhomogeneous nucleation and movement of dislocations. Most of the initial dislocations generated in the molybdenum are not mobile, but are pinned due to the presence of interstitial impurity atoms. Eventually, dislocations are generated which are mobile. The progressive movement and multiplication of dislocations results in the fine slip lines growing into persistant slip bands. Submicroscopic cracks of a few microns form in these slip bands. Klesnil (26) has shown on specimens of low carbon steel that loading to the French damage line followed by reducing the load to the fatigue limit produces no discernable propagation of the submicroscopic cracks. Even after polishing and etching, no visible microscopic cohesion breakdowns were found in the persistent slip bands. However, when the French damage line was exceeded, microscopic cracks of approximately 100 microns were found in the persistent slip bands. Further loading caused propagation of the microscopic cracks that eventually led to fracture of the speciment. Thus, from Klesnil's work, the location of the French damage line in low carbon steel involves localized slip, crack initiation and propagation phenomena. It appears from this work that the French damage line would be more appropriately called a fatigue crack initiation line.

The removal of the upper yield point in molybdenum by cyclic stressing has been qualitatively explained (10) by extending the dynamic dislocation yield model (2,3). The dynamic dislocation yield point model assumes a large number of dislocations are initially pinned by interstitial atoms and only a small number of dislocations are initially mobile. As the magnitude of stress in a monotonic test is increased to the upper yield point, a rapid multiplication of dislocations occur. As the number of these dislocations increase, their velocity decreases and the stress drops. If the initial mobile dislocation density is increased, the magnitude of the yield drop is reduced. If a large number of mobile dislocations are initially present the yield point drop does not occur. Thus the yield point, according to the dynamic dislocation model, depends upon the number of dislocations initially mobile and a rapid dislocation multiplication at the yield point plus the stress dependence of dislocation velocity.

Under cyclic stressing the number of dislocations increase with the number of applied cycles. This increase can spread throughout the entire test section. It may be assumed that a certain percentage of these generated dislocations are not pinned by interstitial atoms but remain mobile. The generation of dislocations by stress cycling can, therefore, provide a sufficient number of mobile dislocations such that the yield drop is eliminated in a subsequent monotonic tensile test. In Fig. 1 it is seen that precycling removed the upper yield point in seven molybdenum specimens. This removal fell within an enclosed region and was dependent upon both the magnitude of alternating stress and number of applied precycles.

The removal of the upper yield point due to cyclic stressing is thus believed to be attributed to localized cumulative multiplications of mobile dislocations. The French damage line, however, appears to best represent the beginning of localized microcrack initiation and propagation. Therefore, even

though the yield point removal line and the French damage line appear to have some macroscopic similarities, two different microscopic phenomena dictate their position. In addition, these lines actually represent an average of a scatter band, particularly the French damage line.

Coaxing

The coaxing experiments contained a prolonged period of cyclic stressing at stress levels below the fatigue limit. The basic mechanism of dislocation movements and generation is identical to that previously described, except that many more cycles are required at the lower stress levels. Due to the diffusion of interstitial atoms to dislocation sites, a larger stress is required to move these dislocations. This is the basic cause of strain aging. During any given stress cycling portion of the coaxing process, certain grains are strengthened by strain aging. This strengthening process continues as the alternating stress level is increased until a stress is reached at which the material can no longer be strengthened by strain aging and the specimen fractures (15).

Several anomalogies occurred in the coaxing study namely: The low fatigue limit of the 2600 F heat treatment and the mixed coaxing response in the 3000 F heat treatment. The low fatigue limit of the 2600 F group appears to be attributed to the increased hydrogen and carbon content after vacuum annealing. These increases are attributed to the vacuum furnace used for the 2600 F anneal. A carbon content ranging from 35 to 44 parts per million in the same bar would be considered normal, however, the 59 ppm would be unreasonable. In addition, most molybdenum of this quality would contain less than 1 ppm of hydrogen. Hydrogen has been shown to reduce the ductility of molybdenum under certain conditions and this effect was found to be more pronounced at intermediate grain sizes (27).

The lack of coaxing response in the 2100 F and 2600 F heat treatments for 10^6 cycle increments can be attributed to insufficient time for proper strain aging at the lower stress levels. The mixed results for the 3000 F

heat treatment can only be attributed to variability and scatter during cyclic stressing. However, it appears the larger grain size in these experiments aided the strain aging behavior at 10^6 cycle increments.

CONCLUSION

Macroscopic similarities exist between the yield point removal line and the French damage line. The basic mechanism involved in these two phenemona, however, is different. The cyclic yield behavior is believed to be dependent upon localized cumulative multiplication of mobile dislocations due to cycling and the French damage line is attributed to localized initiation and propagation of microcracks. More appropriately then, the French damage line might be called a crack initiation line.

The upper and lower yield point in molybdenum is dependent upon grain size. The larger grain sizes cause a decrease in the upper and lower yield point stresses.

Using a cycle ratio summation as a criteria, no definite coaxing pattern occurred for all three molybdenum grain sizes examined. The two smaller grain sizes did not respond to coaxing for all initial stress levels when 10^6 cycle increments were applied. The coarse grain size was susceptible to coaxing for both 10^6 and 10^7 cycle increments. The response however was not consistent. 10^7 cycle increments also caused appreciable coaxing in the smallest grain size at lower initial stresses. Thus, molybdenum does respond to coaxing under certain applied stress and cycle increment conditions.

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TABLE CAPTIONS

FIGURE CAPTIONS

Table 1 Chemical Analysis by Weight Percentage of Molybdenum As-Received

C	O	H	N	Fe	Ni	Si
0.003	0.0004	<0.0001	0.0001	<0.002	<0.001	<0.001

Table 2 Average Properties of Annealed Molybdenum

	Annealing Temperature		
	2150 F 1 hr.	2600 F 1 hr.	3000 F 1 hr.
ASTM Grain Size	7-8	4-5	2-3
Hardness, Rockwell B	88	84	83
Upper Yield Point Stress MN/m^2 (ksi)	572 (83)	345 (50)	324 (47)
Lower Yield Point Stress MN/m^2 (ksi)	469 (68)	289 (42)	274 (40)

Table 3 Interstitial Chemical Content of Molybdenum After Annealing

	Annealing Temperature		
	2150 F 1 hr.	2600 F 1 hr.	3000 F 1 hr.
Carbon	0.0043	0.0059	0.0035
Oxygen	0.0003	0.0002	0.0003
Hydrogen	<0.0001	0.0002	<0.0001
Nitrogen	<0.0001	<0.0001	<0.0001

Table 4 Summary of Coaxing Tests of Molybdenum : Stress Increment = 13.8 MN/m^2 (2 ksi.)

Annealed at 2150 F Fatigue Limit = 327 MN/m^2 (47.5 ksi)

Initial Stress MN/m^2 (ksi.)	Percent of Fatigue Limit	Initial Cycles	Cycle Increment	Stress at Fatigue Failure MN/m^2 (ksi)	$\sum \frac{n}{N}$	Total Cycles to Failure 10^6 Cycles
310 (45)	95	10^6	10^6	338 (49)	.004	2
310 (45)	95	10^6	$2x10^6$	379 (55)	2.1	11
310 (45)	95	10^7	10^6	421 (61)	3.8	18
310 (45)	95	10^7	$2x10^6$	379 (55)	1.7	22
241 (35)	75	10^6	$2x10^6$	400 (57)	3.1	21
241 (35)	75	10^6	10^6	379 (55)	1.0	9
241 (35)	75	10^7	$2x10^6$	379 (55)	2.1	28
159 (23)	50	10^6	10^6	400 (57)	1.5	18
310 (45)	95	10^7	10^7	366 (53)	6.3	40
310 (45)	95	10^7	10^7	366 (53)	7.5	44
276 (40)	85	10^7	10^7	413 (60)	35.4*	103
276 (40)	85	10^7	10^7	427 (62)	44.5*	110
Annealed at 2600 F Fatigue Limit = 190 MN/m^2 (28.5 ksi.)						
186 (27)	95	10^6	10^6	282 (41)	3.0	8
186 (27)	95	10^6	$2x10^6$	241 (35)	1.3	8
186 (27)	95	10^7	10^6	255 (37)	1.1	15
186 (27)	95	10^7	$2x10^6$	214 (31)	.5	13
186 (27)	95	10^7	$2x10^6$	228 (33)	.8	15
145 (21)	75	10^6	10^6	255 (37)	1.1	9
145 (21)	75	10^6	$2x10^6$	282 (41)	6.5	20
97 (14)	50	10^6	10^6	234 (34)	.5	11
97 (14)	50	10^6	$2x10^6$	248 (36)	1.4	22
Annealed at 3000 F Fatigue Limit = 276 MN/m^2 (40 ksi.)						
262 (38)	95	10^6	10^6	317 (46)	1.4	5
262 (38)	95	10^7	10^7	358 (52)	173*	80
241 (35)	87.5	10^6	10^6	420 (61)	808*	14
241 (35)	87.5	$2x10^7$	10^7	296 (43)	5	61
241 (35)	87.5	10^7	10^7	324 (47)	13	65
205 (30)	75	10^6	10^6	427 (62)	740*	17
205 (30)	75	10^7	10^7	372 (54)	190*	122

* Indicates Appreciable Coaxing Effect

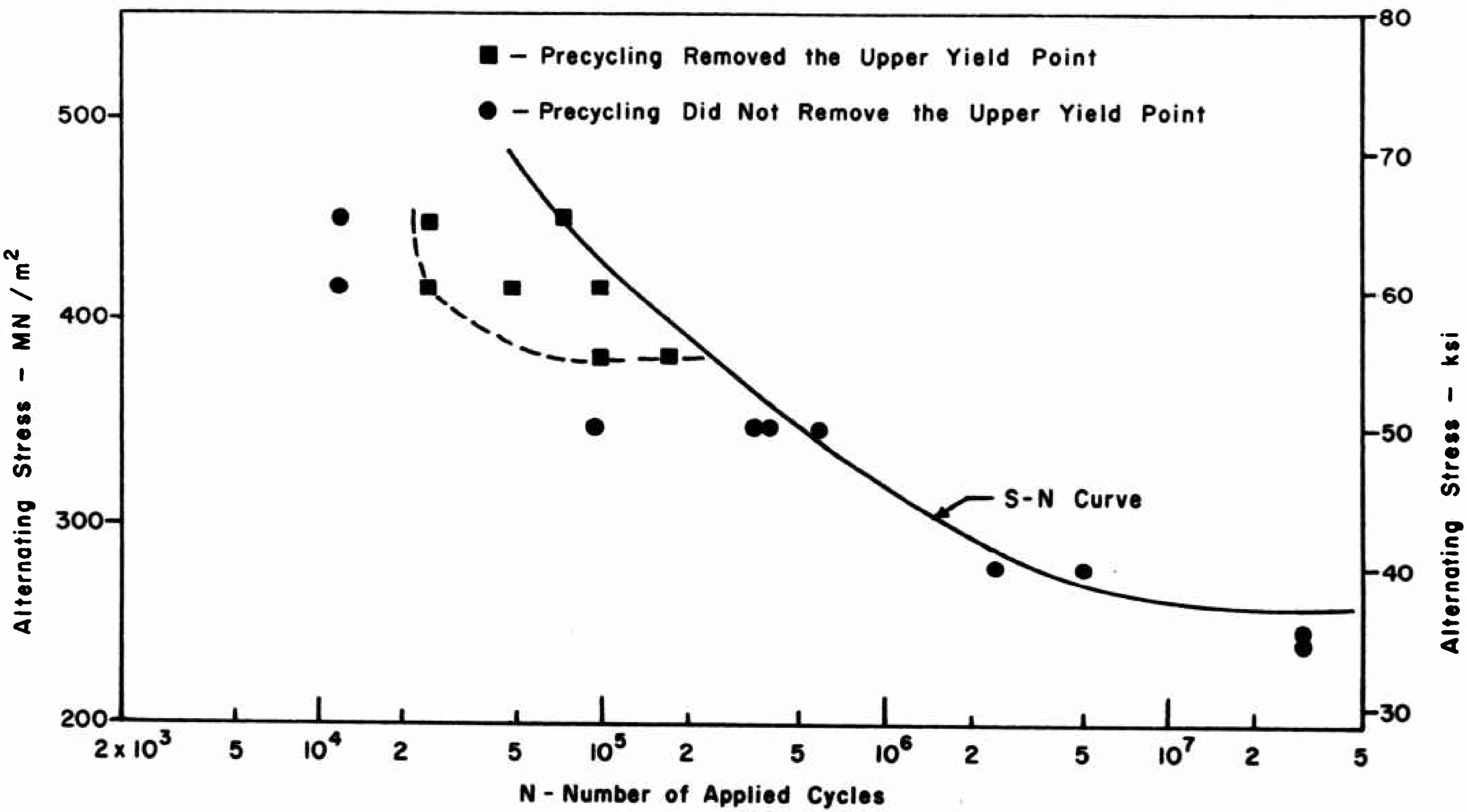


Fig. 1 Cyclic Yield Behavior and Fatigue of Molybdenum Annealed at 2100° F., Fully Reversed Axial Loading (ref. 10)

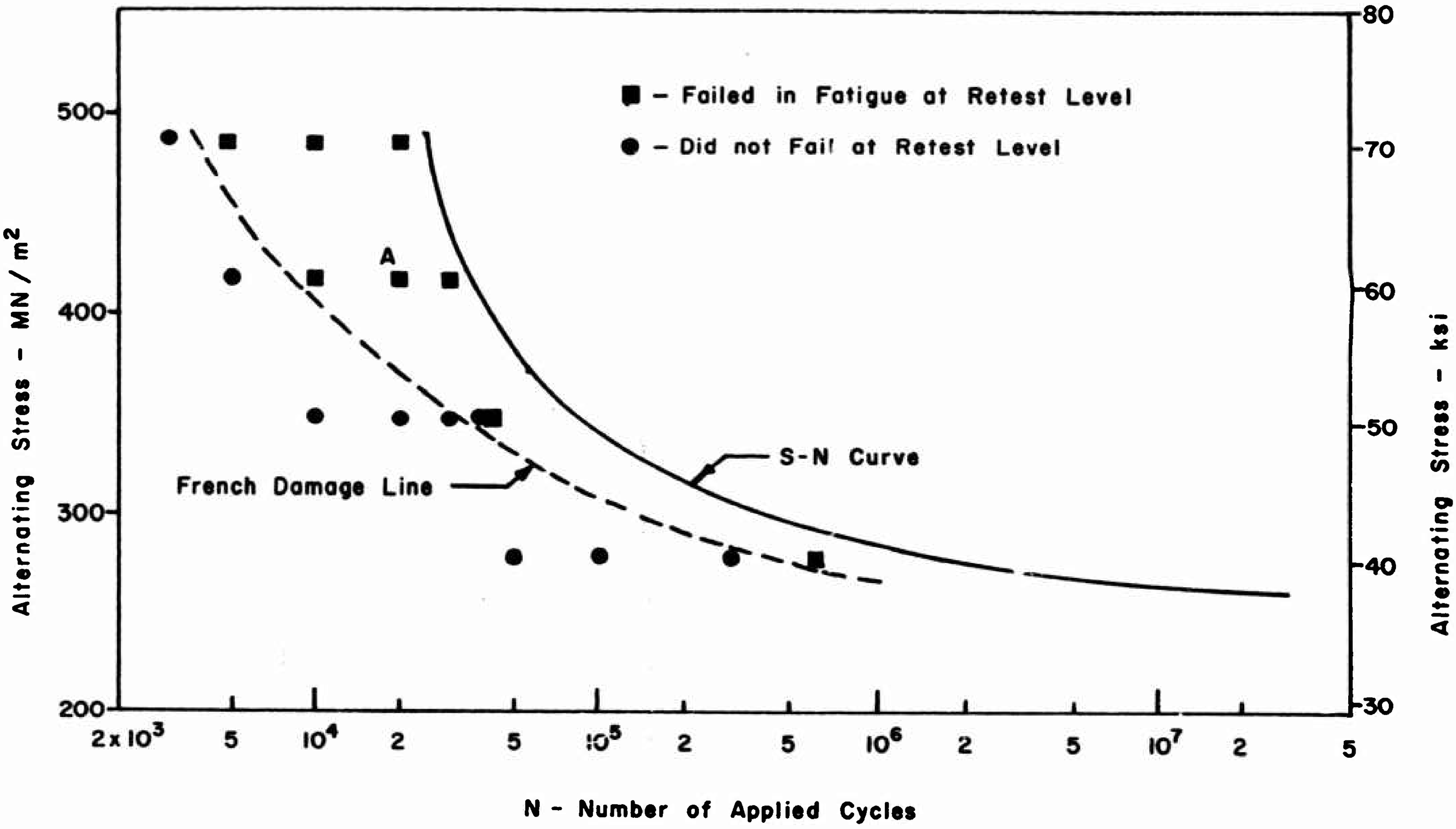


Fig. 2 French Damage Line for Molybdenum Annealed at 2150° F., Fully Reversed Axial Loading

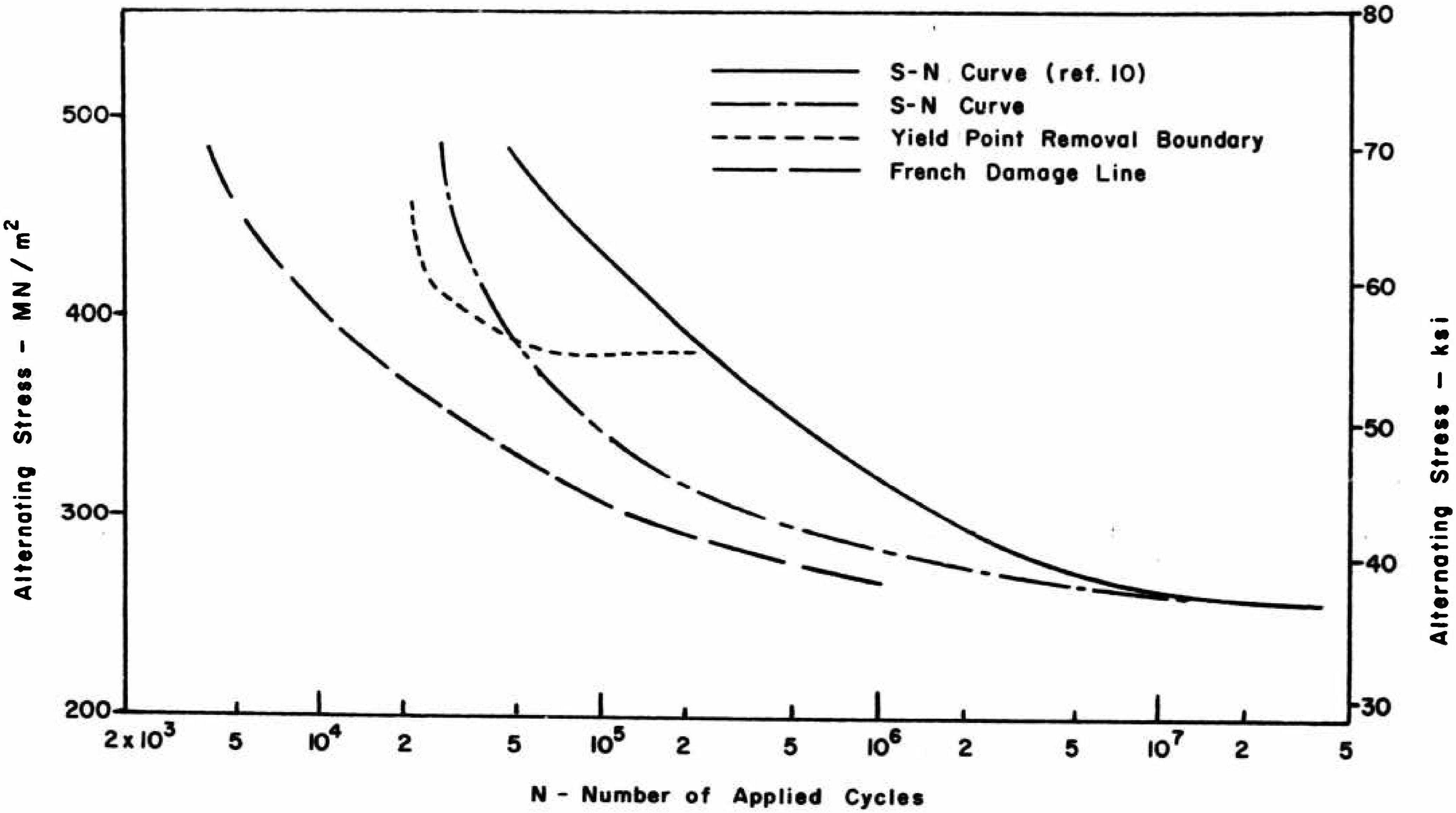
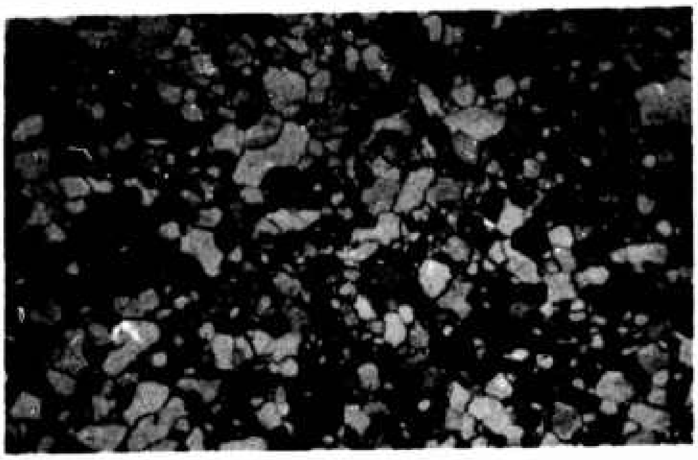


Fig. 3 Comparison of Cyclic Yield Behavior and French Damage Line in Molybdenum, Fully Reversed Axial Loading



(a) 2150° F. for 1 hour X100

(b) 2600° F. for 1 hour X100

(c) 3000° F. for 1 hour X100

Fig. 4 Grain Size of Annealed Molybdenum

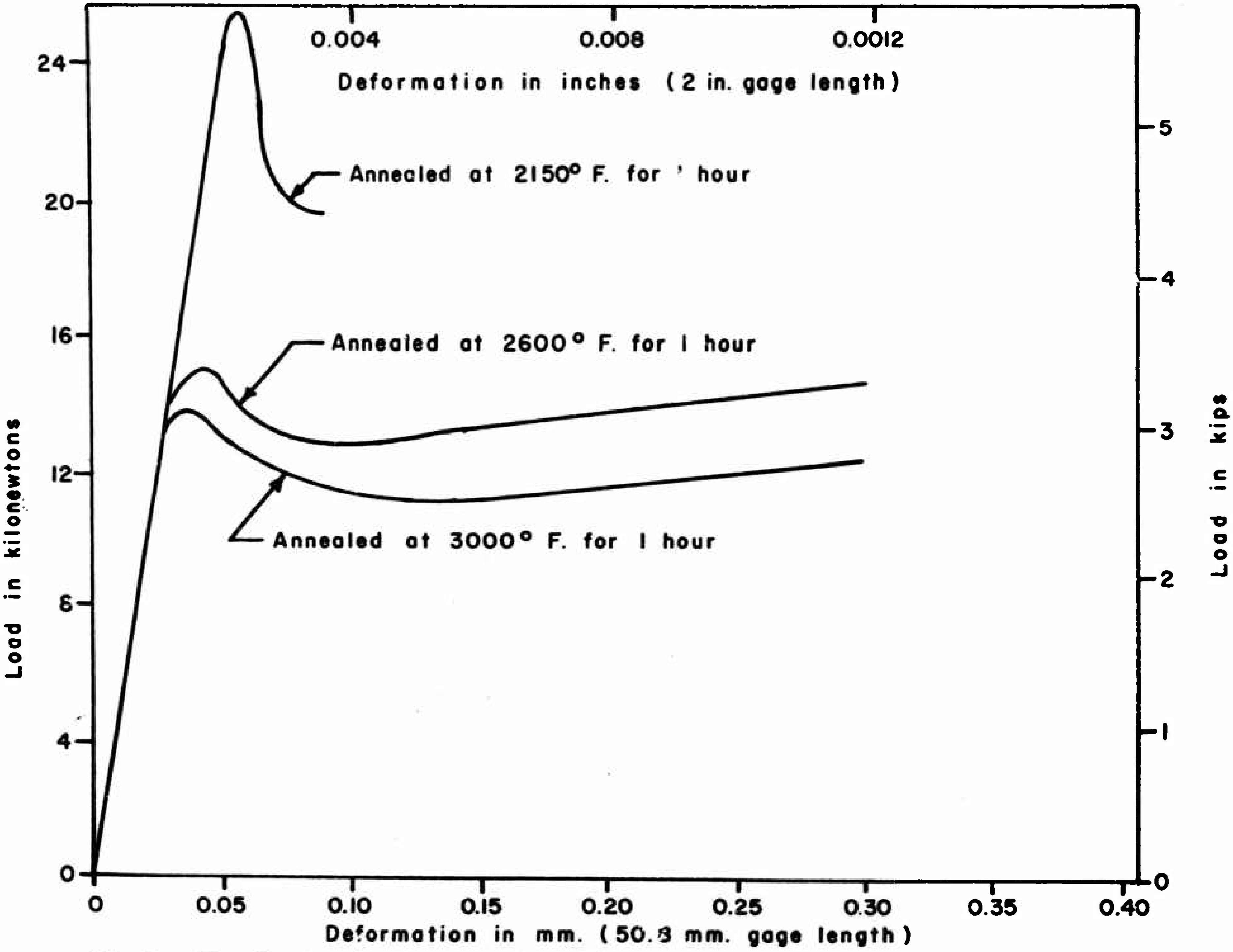


Fig. 5 Tensile Load - Deformation Diagrams for Annealed Molybdenum

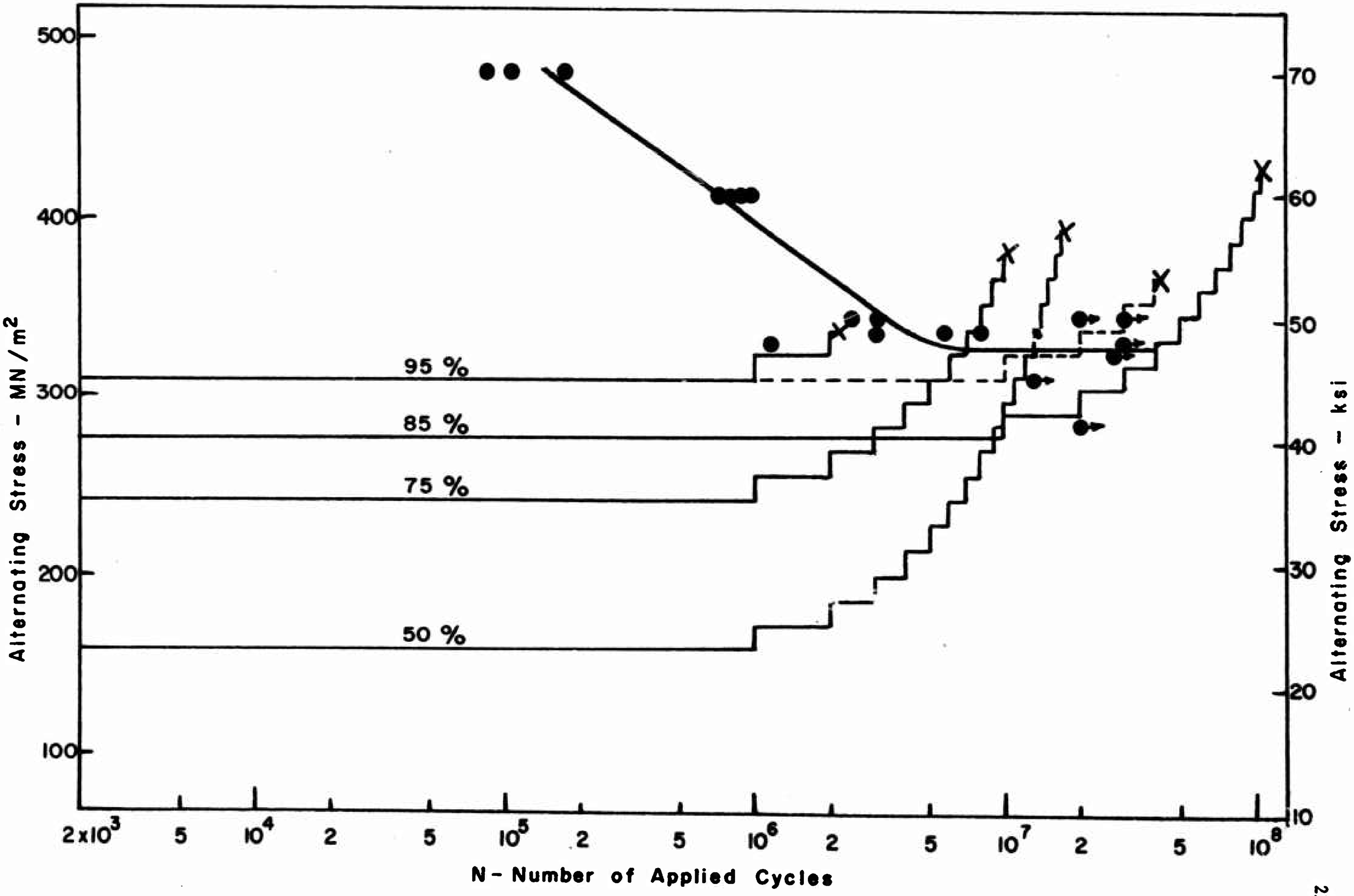


Fig. 6 S-LogN Diagram and Coaxing of Molybdenum Annealed at 2150° F., Rotating Bending R = -1

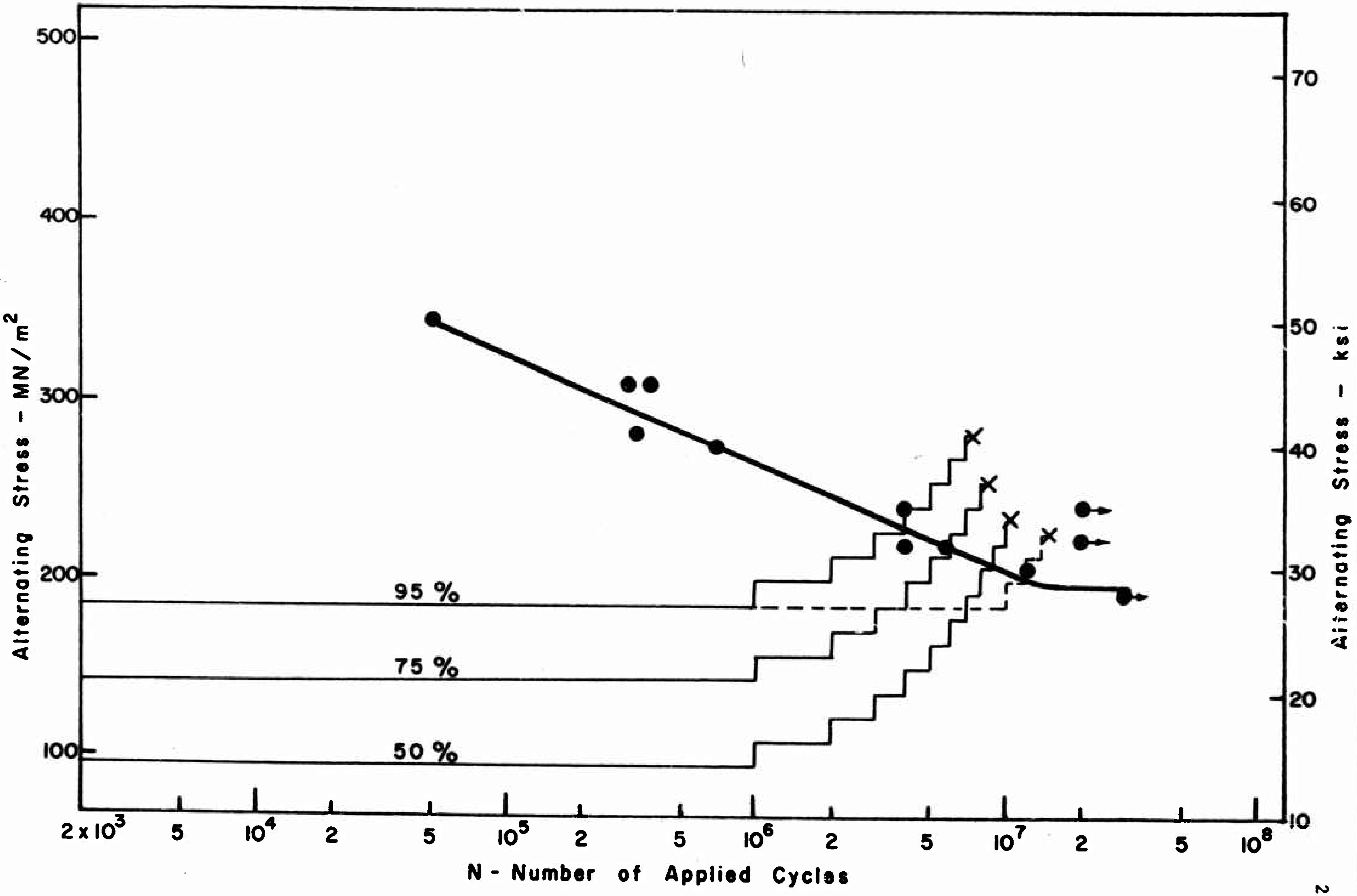


Fig. 7 S - LogN Diagram and Coaxing of Molybdenum Annealed at 2600° F., Rotating Bending R = -1

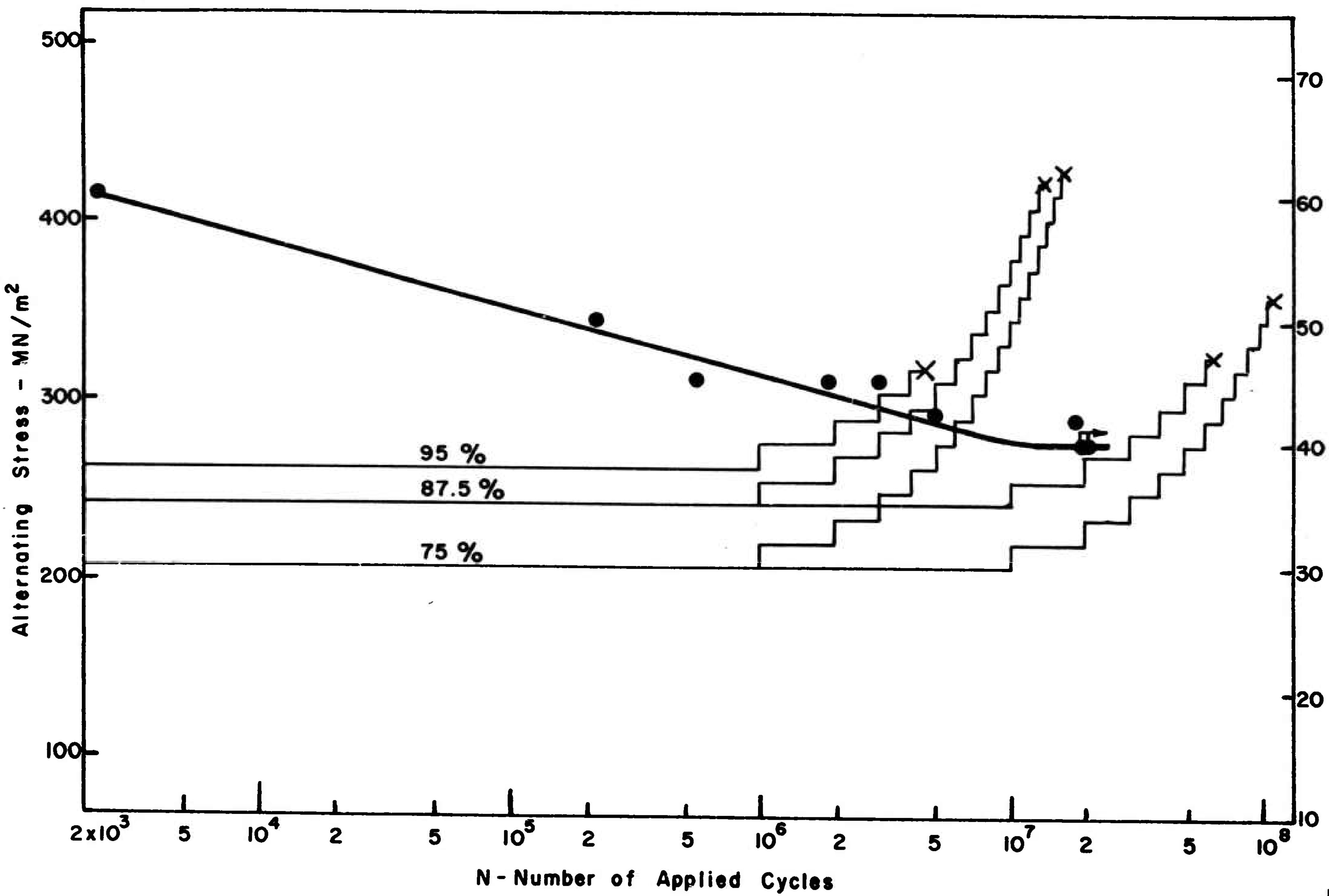
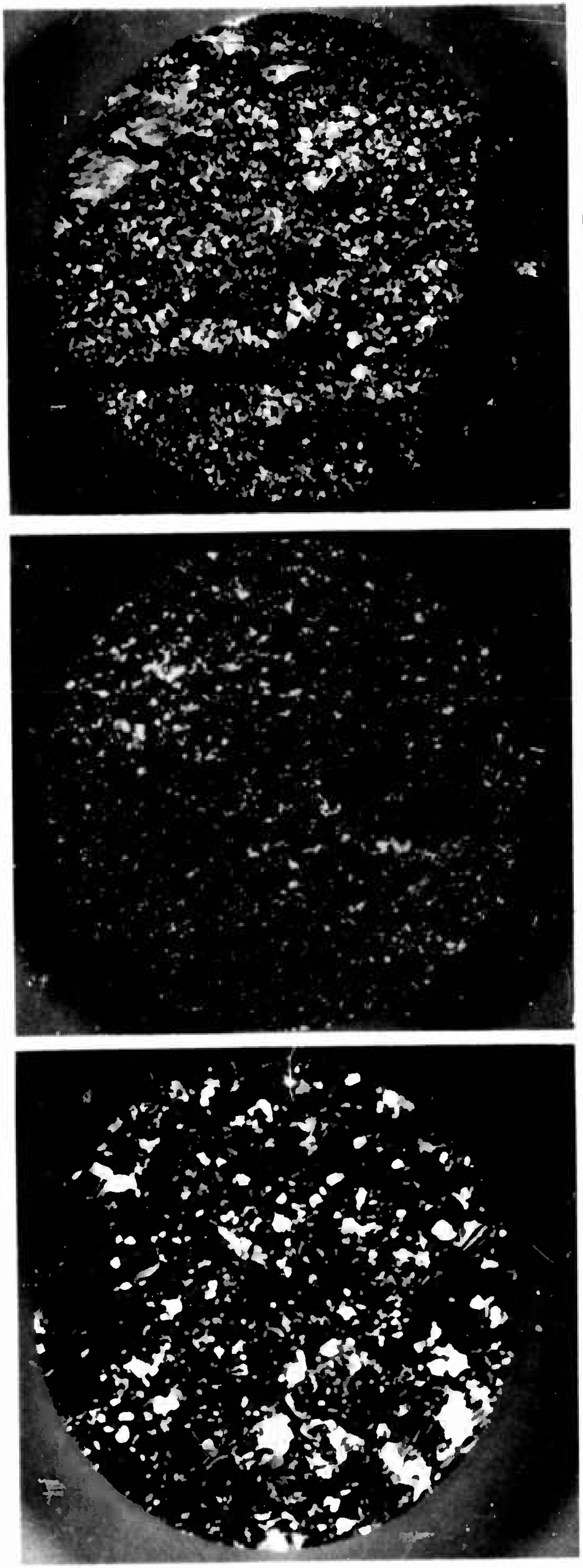


Fig. 8 S - LogN Diagram and Coaxing of Molybdenum Annealed at 3000° F., Rotating Bending R = -1



(a) Annealed at 2150° F. for 1 hour. X16

(b) Annealed at 2600° F. for 1 hour. X16

(c) Annealed at 3000° F. for 1 hour. X16

Fig. 9 Fracture Surfaces of Annealed Molybdenum After Applied Coaxing, Rotating Bending R = -1.

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13. ABSTRACT

The objective of this research was to further elucidate the fatigue behavior of commercially pure arc-cast polycrystalline molybdenum; in particular, to investigate the possible relationship between the French damage line and cyclic dependent yield behavior, and to determine coaxing phenomena as a function of grain size. Although the French damage line and the cyclic yield point removal line appear macroscopically similar, the mechanisms proposed for these two phenomena are quite different. The removal of the upper yield point under cyclic stress conditions is explained in terms of localized cumulative multiplication of mobile dislocations, whereas the French damage line is explained in terms of the initiation and propagation of microcracks. Molybdenum, vacuum annealed at 2150 F, 2600 F and 3000 F with resulting ASTM grain sizes of 7-8, 4-5 and 2-3 respectively, was subjected to various coaxing patterns. Using a cycle ratio summation as a criteria, no definite coaxing response pattern occurred for all three molybdenum grain sizes. Cycle ratio summations $\Sigma\ n/N$ ranged from .004 to 808. Thus molybdenum did respond to coaxing under certain applied stress and cycle increment conditions.

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damage						
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cumulative damage						
crack initiation						
yielding						
molybdenum						